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(54) PROCESSES FOR PRODUCING VITREOUS FIBRES

(71) We, JAPAN INORGANIC MATERIAL CO., LTD., a Japanese Body Corporate, of No. 1044—11, Fugisawa, Fugisawa-shi, Kanagawa-ken, Japan, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention relates to processes for producing vitreous fibres.

This invention provides a process for producing vitreous fibres, in which process a powder, comprising from 35 to 75% (calculated as silica) of a silicaceous material, from 10 to 55% (calculated as calcium oxide) of a calciferous material, minor amounts of metal oxides, and unavoidable impurities, is fused to form a melt which is passed through orifices or spinnerets to form the desired vitreous fibres.

Thus, silica (SiO_2) and calcium oxide (CaO) are effectively the two principal ingredients of the powder, but it also contains smaller amounts of oxides, and by the term "minor amounts of metal oxides" is meant amounts of from 2 to 13% of alumina (Al_2O_3), from 0 to 10% of zirconium oxide (ZrO_2), from 0 to 4% of ferric oxide (Fe_2O_3), from 0 to 3% of sodium oxide (Na_2O), from 0 to 3% of potassium oxide (K_2O), from 0 to 2% of magnesium oxide (MgO) and from 0 to 1% of titanium oxide (TiO_2).

All percentages quoted in the Specification refer to percentages by weight.

The powder could be made up by mixing together the required amounts of the various oxides — thus, by mixing suitable amounts of silica calcium oxide, alumina, ferric oxide and so on. However, many of the oxides themselves are very reactive, and as it is often most convenient to add the silicaceous material in the form of silicates and/or aluminosilicates. Such compounds are particularly convenient since they are widely available; a most suitable source of material for the powder used in the process of the invention is portland cement (known also as white cement), which contains silicon, calcium, aluminium and iron in the following amounts (calculated, as are all percentage compositions quoted hereinafter, in terms of their oxide):—

SiO_2	20 to 32%;
CaO	60 to 72%;
Al_2O_3	1 to 5%; and
Fe_2O_3	1 to 6%.

It is, therefore, preferable to make up the powder to be used in the process of the invention by mixing portland cement with a silica-rich material to give the desired composition.

Portland cement (which is commonly termed simply "cement", and will be referred to as such hereinafter) is produced by grinding limestone and clay to a fine powder, forming this powder into a thick slurry with water, and firing it in a kiln. At a temperature of about 1500° C, the aluminosilicates from the clay are sintered to form small balls called clinkers. The clinkers are then crushed, for example in a ball mill, to give the fine powder known as cement. It is normal practice then to add small amounts of gypsum ($CaSO_4 . 2H_2O$) to the cement — typically up to 2% of gypsum is added.

However, gypsum decomposes to give sulphur trioxide (SO_3) gas at the temperature used in vitreous fibre manufacture, and the production of any gas is undesirable since it may lead to the production of bubbles in the fibres. Consequently it is advantageous, though not essential, to use in the process of the invention cement containing substantially no gypsum or other material capable of releasing SO_3.

The advantages of using cement as a starting material to make vitreous fibres are:

a) it is readily available in large quantities all over the world;

b) it is stable if kept dry;

c) it is available in the right form — a fine powder;

d) it is cheap.

However, until now, it has proved virtually impossible to prepare vitreous fibres simply by heating cement in a manner analagous to

the conventional preparations of vitreous fibres (in which a suitable glass is heated to from 1100 to 1500° C, and formed into the desired fibres). However, by the addition of silica-rich material to the cement a powder may be formed from which vitreous fibres may be prepared. By the term "silica-rich material" as used herein is meant a material which contains at least 32.6% (calculated as silica) of silicon. Naturally, the higher the silica content of this silica-rich material, the smaller the amount that has to be added to the cement to give a powder having a silica content in the required range; therefore preferably the silica-rich material contains greater than 60% (calculated as silica) of silicon. Silica itself can be used; commercial silica powder is readily available, and contains from 98 to 99% silica. However, there are many naturally-occurring silica-rich materials which may be used, and these are preferred because of their lower cost and ready availability. Examples of such materials are silica sand (97 to 98% silica), certain white clays (approximately 95% silica), shirasu (SiO_2 65 to 73%, Al_2O_3 12 to 16%, Fe_2O_3 1 to 3%, CaO 2 to 4%, K_2O 2 to 4%, Na_2O 3 to 4%), which is available in large quantities in Kagoshima Prefecture, Japan, and zircon flour (SiO_2 32.60%, ZrO_2 66.90%, TiO_2 0.12%, Al_2O_3 0.43%, Fe_2O_3 0.04%, MgO 0.03%, P_2O_5 0.007%).

The composition of the invention may contain one or more of the above-described silica-rich materials, and, by way of general illustration, it may be said that powders having a composition in the following ranges have been found particularly effective:—

a) 50 to 80% cement and
   50 to 20% silica sand;
b) 20 to 55% cement and
   45 to 80% shirasu;
c) 20 to 55% cement,
   20 to 40% shirasu and
   20 to 40% silica sand;
d) 40 to 50% cement,
   35 to 47.5% silica sand and
   2.5 to 25% zircon flour;
e) 40 to 50% cement,
   35 to 48% shirasu and
   2.5 to 25% zircon flour; and
f) 40 to 50% cement,
   18 to 25% silica sand,
   17 to 25% shirasu and
   2.5 to 25% zircon flour.

It has also been found that the production of vitreous fibres is in general greatly facilitated by having the silica-rich material of substantially the same particle size as the cement, since this ensures good mixing of the two components when the powder is melted.

The silicon content of the powder used in the process of the invention is preferably from 45 to 65% (calculated as silica), and most preferably from 50 to 60% (calculated as silica).

The powder is formed into fibres by a spinning operation — that is, it is heated until it is molten, and then passed through orifices or spinnerets to form vitreous fibres. The temperature to which the powder has to be heated so that it melts is, of course, dependent on its composition. It has been found that the spinning operation is most easily performed at a temperature of from 1100 to 1500° C, and thus for convenience the powders should melt well within this range. Powders that melt at temperatures greater than 1500° C give rise to difficulties; they tend to solidify quickly, and this prevents the formation of long fibres. Preferably the powder used in the process of the invention has a melting point of from 1200 to 1300° C.

The process of the invention may be used to prepare either long or short vitreous fibres. By way of illustration only, two spinning apparatuses will now be described with reference to the accompanying drawings, in which:—

Figures 1 and 2 are schematic drawings of apparatuses for producing short and long fibres respectively, which may be employed in the process of the invention.

Figure 1 shows two views of a typical apparatus for producing short fibres; Figure 1A is a front view and Figure 1B is a side view. The powder containing cement and silica-rich material is introduced into the melting furnace (1) where it is heated to a temperature at which it is completely melted. The formed melt flows out through orifices in the bottom of the furnace to form single fibres (2), which are supported by a comb (3), and separated so that none of the fibres cross or become tangled. The fibres are formed into the required thickness by passage between rubber rollers (4), and are then cut to length with a burner (5).

The maximum temperature of the furnace is 1500° C; powders that melt at a temperature higher than 1500° C, such as some powders containing iron, are not used since it is difficult to obtain single fibres. For safety reasons, it is desirable if the furnace is operated at a temperature not higher than 1500° C, and preferably in the range of from 1200 to 1300° C.

Figure 2 shows two sides of a typical apparatus for producing long fibres. Figure 2A is a front view and Figure 2B is a side view. The powder containing cement and silica-rich material is introduced into the furnace (1) where it is heated to a temperature at which it is completely melted. The melt is then passed through orifices to form single fibres (2). Several single fibres, to which a binding agent is applied from an applicator (6), are united by a grooved roller (7) to form a strand (8). The formed strand is then wound

at high speed onto the winding drum (10), the strand being moved across the drum by means of a traverse guide (9).

As with the apparatus for producing short fibres, the maximum practical temperature of the furnace in this apparatus is 1500° C.

The following Examples are now given, though only by way of illustration, to show materials, conditions and techniques which may be employed in the process of the invention. In these Examples where mesh sizes are quoted they refer to the JIS standard of mesh sizes.

Example 1.

Production of long vitreous fibres from cement and silica sand.

a) Starting materials

Commercially available portland cement (containing small amounts of gypsum) was formed into a powder with silica sand. Finely powdered (to pass through a 65-mesh sieve) silica-rich material which had been converted into tridymite or cristobalite was employed. Suitable materials are, for example, silica sand from Yuo Island, white clay from Beppu, Japan or commercial silica powder, the chemical compositions of which are shown in the following Table.

Chemical Composition of silica-rich materials

	SiO_2	Al_2O_3	Fe_2O_3	TiO_2	CaO	MgO	K_2O	Na_2O
Silica sand from Yuo Island	97.50	0.88	0.014	0.29	0.02	0.10	0.04	0.18
White Clay from Beppu	95.25	1.36	0.12	1.11	0.16	0.08	0.02	0.14
Commercial silica powder	98.62	0.51	0.044	0.02	0.07	0.05	0.06	0.03

b) Process

The cement/silica powder was first thoroughly mixed in a ball mill and then introduced into a furnace where it was heated to a temperature of 1450° C at which it was completely melted. The formed melt flowed out from nozzles of 4mm in diameter, and the fibres formed were wound onto a drum while being maintained at an appropriate temperature. The fibres obtained were subjected to tests, and the results are summarised in Table 1 below.

When a composition lying outside the range of compositions shown in the Table was used, spinning was impossible at a temperature below 1500°. For example, when the ratio in percentage by weight of the portland cement to the silica powder (selected from the group consisting of white clay from Beppu, Silica sand from Yuo Island and commercial silica powder) was greater than 8/2, the spinning temperature exceeded 1500° C.

TABLE 1

Powder	Composition									Spinning temperature (°C)	Diameter of fibre μ	Tensile strength (kg/mm^2)	Elongation (%)
	SiO_2	Al_2O_3	Fe_2O_3	TiO_2	CaO	MgO	K_2O	Na_2O	SO_3				
Ratio of Portland cement to Silica sand (from Yuo Island)													
5:5	59.55	3.04	1.607	0.145	32.61	0.9	0.02	0.09	0.75	1390 – 1440	18.025	68.369	2.00
6:4	51.96	3.472	1.926	0.116	39.13	1.06	0.016	0.072	0.90	1410 – 1440	23.908	52.278	2.275
7:3	44.37	3.904	2.244	0.087	45.646	1.22	0.012	0.054	1.05	1410 – 1440	23.424	59.682	1.325
8:2	36.87	4.336	2.563	0.058	52.164	1.38	0.008	0.036	1.20	1440 – 1490	20.132	72.400	2.225
Ratio of Portland cement to White clay (from Beppu)													
5:5	58.43	3.28	1.66	0.555	32.68	0.89	0.01	0.07	0.75	1390 – 1440	33.533	60.283	2.920
6:4	51.06	3.66	1.968	0.444	39.18	1.05	0.008	0.056	0.90	1390 – 1440	29.683	45.460	2.750

TABLE 1 (Continued)

Powder	Composition	Spinning temperature	Diameter of fibre	Tensile strength	Elongation

TABLE 1 (Continued)

Powder	Composition									Spinning temperature (°C)	Diameter of fibre μ	Tensile strength (kg/mm$_2$)	Elongation (%)
	SiO_2	Al_2O_3	Fe_2O_3	TiO_2	CaO	MgO	K_2O	Na_2O	SO_3				
Ratio of Portland cement to Commercial silica powder													
5:5	60.11	2.86	1.622	0.01	32.64	0.835	0.03	0.015	0.75	1440	23.518	43.549	2.500
6:4	52.41	3.32	1.938	0.008	39.128	1.04	0.024	0.012	0.90	1420 – 1480	30.310	56.179	2.775
7:3	44.71	3.19	2.253	0.006	45.661	1.21	0.018	0.009	1.05	1380 – 1460	19.445	63.956	1.650
8:2	37.00	4.26	2.569	0.004	52.174	1.37	0.012	0.006	1.20	1440 – 1450	19.002	52.728	1.900

Example 2.

Production of long vitreous fibres from cement and shirasu.

a) Starting Material

A powder was prepared by mixing cement and shirasu. The cement used was the same as that used in Example 1. Shirasu is a naturally occurring material, found in large amounts in Kagoshima Prefecture, Japan, for which, until now, there has been no particularly profitable use, and it is therefore a very inexpensive starting material. The compositions of a number of samples of shirasu are given in Table 2; the shirasu used in this Example had the composition of sample 10, from which any iron was first removed with a magnet.

TABLE 2

Place of occurrence	Aira caldera					
Sample number	1	2	3	4	5	6
Designation	Aira shirasu	,,	,,	Aira secondary (sedimantary shirasu)	Aira shirasu	,,
Sampling place	Kagoshima city	,,	,,	,,	Nakagawa Koriyamacho, Hiokigun	,,
SiO_2	68.60	71.02	71.06	71.40	70.40	71.22
TiO_2	0.29	0.20	0.26	0.25	0.29	0.25
Al_2O_3	15.08	14.67	13.62	13.65	13.96	13.51
Fe_2O_3	0.98	1.19	0.52	0.76	0.86	0.89
FeO	1.80	1.56	1.71	1.55	1.67	1.57
MnO	0.07	0.07	0.05	0.04	0.05	0.05
MgO	0.61	0.60	0.61	0.48	0.64	0.54
CaO	2.66	2.71	2.17	1.88	2.06	2.19
Na_2O	3.44	3.20	3.30	3.36	3.06	3.50
K_2O	2.28	2.28	2.70	2.70	2.70	2.82
H_2O+	3.04	2.42	3.12	3.40	3.14	2.84
H_2O-	0.50	0.44	0.48	0.62	0.52	0.36
P_2O_5	0.08	0.10	0.10	0.05	0.09	0.09
Total	99.43	100.46	99.70	100.14	99.44	99.83

TABLE 2 (continued)

7	8	9	10	11	12	13	14	15
Aira caldera					Ikeda caldera	Ata caldera		

TABLE 2 (continued)

Aira caldera								Ikeda caldera	Ata caldera	Ata caldera
7	8	9	10	11	12	13	14	15	16	17
Aira Shirasu	,,	,,	,,	,,	,,	,,	Egnchima Higashi Ichiki-cho Hioki-gun	Ikeda-shirasu	Water-coloured matter at lower part of Ata shirasu	Brown-coloured matter at upper part of Ata shirasu
Nakano Kajiki-cho Aira-gun	Izumi city	Sanmonji Osaki-cho Soo-gun	Shibushicho Soo-gun Soo-gun	Makurazaki city	,,	Sakanoue Taniyama city	,,	Ikezaki Ikeda-ko	Onejime-cho Kimotsuki-gun	Onejime-cho Kimotsuki-gun
68.48	71.86	71.70	70.82	72.54	71.46	71.72	68.07	69.76	64.67	66.06
0.26	0.20	0.20	0.24	0.28	0.23	0.21	0.36	0.43	0.62	0.66
14.74	13.35	13.59	14.02	13.56	14.15	14.14	16.13	13.82	15.44	15.43
1.23	0.93	0.75	0.95	0.52	0.54	0.64	1.09	1.57	4.72	2.82
1.59	1.38	1.47	1.40	1.51	1.68	1.51	1.41	1.71	1.82	1.47
0.37	0.05	0.05	0.07	0.08	0.08	0.05	0.04	0.02	0.10	0.10
0.47	0.52	0.62	0.52	0.48	0.44	0.48	0.97	0.99	0.98	0.87
2.64	1.92	2.07	1.90	1.96	1.99	2.13	2.90	0.69	3.47	3.55
2.90	3.30	3.52	3.30	3.30	3.72	3.20	3.21	3.72	3.47	3.20
2.10	2.70	2.90	2.90	2.54	2.28	2.48	2.98	2.48	1.58	1.80
4.54	3.42	3.08	3.60	2.60	2.96	2.92	2.83	2.30	3.11	3.00
1.14	0.44	0.48	0.96	0.36	0.38	0.50	0.68	0.44	0.53	0.46
0.07	0.07	0.06	0.05	0.02	0.01	0.04	0.14	0.09	0.13	0.16
100.53	100.34	100.49	100.77	99.75	99.92	100.02	100.81	100.02	100.64	99.58

b) Process

The cement/shirasu powder was introduced into a furnace and heated to a temperature of 1440° C at which it was completely melted. The melt was passed through orifices of 4mm diameter to form fibres, which were wound onto a drum while being maintained at an appropriate temperature. The fibres obtained were tested, and the results are shown in Table 3.

As in Example 1, if a powder was employed having a composition outside the range of composition shown in Table 3, spinning was impossible at a temperature below 1500° C. Thus, the spinning temperature became higher than 1500° C when the ratio of portland cement to "shirasu" lay outside the range of from 55:45 to 20:80.

TABLE 3

Admixing ratio by weight of portland cement to "shirasu"	Load (g)	Tensile strength (kg/mm²)	Elongation (%)	Diameter of fibre (μ)	Composition						
					SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	K_2O	Na_2O
55/45	17.33	40.167	1.023	15.825	42.93	9.16	2.66	37.21	0.94	1.3	1.58
50/50	18.05	58.667	1.625	15.724	45.30	9.60	2.60	34.10	0.85	1.5	1.75
45/55	16.96	55.526	1.598	15.904	47.67	10.04	2.54	30.99	0.77	1.6	1.93
40/60	15.88	52.705	1.575	16.001	50.04	10.48	2.48	27.88	0.68	1.8	2.10
35/65	12.01	47.522	1.336	14.022	52.41	10.92	2.42	24.77	0.60	1.9	2.22
30/70	7.94	41.866	1.075	12.588	54.78	11.36	2.36	21.66	0.51	2.1	2.45
20/80	8.25	40.350	1.005	12.010	57.38	12.24	2.20	18.32	0.38	2.4	2.74

Example 3.

Production of long vitreous fibres from cement, silica sand and zircon flour.

a) Starting materials

Powders were formed containing equal amounts (by weight) of cement and

(i) silica sand from Yuo Island, or

(ii) commercial silica powder.

The particle size of each of these powders was such that the powders passed through a 65-mesh sieve.

To the above mixtures, zircon flour (weight ratio of $SiO_2 : ZrO_2$ 36:44) which passed through a 300-mesh sieve was added in amounts of 2.5, 7.5, 12.5, 17.5 and 25.0% of the mixture.

b) Process

The above powders were thoroughly mixed in a ball mill, and then introduced into a furnace and heated to a temperature of 1490° C at which the mixtures were completely melted. The formed melt was passed

through orifices to form fibres, which were wound onto a drum while being maintained at an appropriate temperature.

The fibres obtained were tested, and the results are shown in Table 4.

TABLE 4

...was such that the ... each of these powders 65-mesh sieve. ...owders passed through a

...above powders were thoroughly mixed in a ball mill, and th... introduced into a furnace and heated to a temperature of 1490° C at which the mixtures were completely melted. The formed melt was passed through orifices to form fibres, which were wound onto a drum while being maintained at an appropriate temperature.

The fibres obtained were tested, and the results are shown in Table 4.

TABLE 4

	Zircon flour %	Diameter of fibre (μ)	Tensile strength (Kg/mm^2)	Composition (% by weight)								
				SiO_2	Al_2O_3	Fe_2O_3	TiO_2	ZrO_2	CaO	MgO	K_2O Na_2O	SO_3
Cement 50% + Silica sand (from Yuo Island) 50%	2.5	18.43	64.06	59.15	3.01	1.53	1.14	1.00	32.13	0.92	0.10	0.74
	7.5	20.26	67.53	58.33	2.90	1.54	0.14	3.04	31.13	0.86	0.10	0.72
	12.5	18.51	68.53	57.51	2.84	1.49	0.14	5.07	30.14	0.83	0.09	0.69
	17.5	19.30	71.41	56.69	2.76	1.44	0.14	7.09	29.15	0.81	0.09	0.67
	25.0	18.09	64.46	56.44	2.64	1.37	0.14	10.14	27.65	0.77	0.08	0.64
Cement 50% + Silica Powder 50%	2.5	14.14	69.42	59.70	2.82	1.60	0.01	1.01	32.15	0.86	0.04	0.74
	7.5	14.97	70.28	58.86	2.74	1.55	0.02	2.03	31.15	0.84	0.04	0.72
	12.5	16.48	75.11	58.02	2.66	1.50	0.02	5.06	30.16	0.81	0.04	0.69
	17.5	15.92	82.69	57.19	2.60	1.46	0.02	7.08	29.18	0.79	0.04	0.67
	25.0	17.19	68.17	55.91	2.49	1.39	0.02	10.11	29.67	0.75	0.04	0.64

Example 4.

Production of short vitreous fibres from cement, shirasu and silica sand.

a) Starting material

Portland cement containing no gypsum was formed into a powder with from 20 to 40% of shirasu and from 20 to 40% of silica sand.

b) Process

The cement/shirasu/silica powder was introduced into a furnace and heated to a temperature of 1440° C at which it was completely melted. The formed melt was passed through orifices to form the desired fibres, and these fibres were cut into short lengths with a burner.

c) Results

Physical properties of the short fibres produced:

Appearance: White, bulky
Diameter: less than 3μ
Length: 5 to 10 mm
Content of granular substances: less than 3%

As may be seen from all of the above Examples the process of the present invention makes it possible to form vitreous fibres having excellent characteristics from readily available starting materials.

WHAT WE CLAIM IS:—

1. A process for producing vitreous fibres, in which process a powder comprising from 35 to 75% (calculated as silica) of a siliceous material, from 10 to 55% (calculated as calcium oxide) of a calciferous material, minor amounts of metal oxides (as hereinbefore defined), and unavoidable impurities, is fused to form a melt which is passed through orifices or spinnerets to form the desired vitreous fibres.

2. A process as claimed in claim 1, in which the silicaceous material is in the form of silicates and/or aluminosilicates.

3. A process as claimed in claim 1, in which the powder comprises portland cement and a silica-rich material (as hereinbefore defined).

4. A process as claimed in claim 3, in which the cement contains substantially no gypsum or other material capable of releasing SO_3.

5. A process as claimed in either of claims 3 and 4, in which the silica-rich material contains greater than 60% (calculated as silica) of silicon.

6. A process as claimed in any of claims 3 to 5, in which the silica-rich material is one or more of silica sand, white clay, shirasu and zirconflour mix.

7. A process as claimed in claim 6, in which the powder contains 50 to 80% cement and 50 to 20% silica sand.

8. A process as claimed in claim 6, in which the powder contains 20 to 55% cement and 45 to 80% shirasu.

9. A process as claimed in claim 6, in which the powder contains 20 to 55% cement, 20 to 40% shirasu and 20 to 40% silica sand.

10. A process as claimed in claim 6, in which the powder contains 40 to 50% cement, 35 to 47.5% silica sand and 2.5 to 25% zirconflour.

11. A process as claimed in claim 6, in which the powder contains 40 to 50% cement, 35 to 48% shirasu and 2.5 to 25% zirconflour.

12. A process as claimed in claim 6, in which the powder contains 40 to 50% cement, 18 to 25% silica sand, 17 to 25% shirasu and 2.5 to 25% zirconflour.

13. A process as claimed in any of claims 3 to 12, in which the silica-rich material has substantially the same particle size as the cement.

14. A process as claimed in any of the preceding claims, in which the silicon content of the powder is from 45 to 65% (calculated as silica).

15. A process as claimed in claim 14, in which the silicon content is from 50 to 60% (calculated as silica).

16. A process as claimed in any of claims 7 to 12, in which the powder melts in the range of 1100 to 1500° C.

17. A process as claimed in claim 16, in which the powder melts at from 1200 to 1300° C.

18. A process as claimed in claim 1, and substantially as described hereinbefore.

19. Vitreous fibres whenever prepared by a process as claimed in any of the preceding claims.

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20 to 55%
and 20 to 40%

in claim 6, in
to 50% cement,
2.5 to 25%

in claim 6, in
to 50% cement,
to 25% zircon-

claim 6, in
50% cement,
% shirasu and

any of claims
material has
size as the

any of the
con content
(calculated

im 14, in
50 to 60%

of claims
in the

16, in
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